NON DESTRUCTIVE 3D X-RAY IMAGING OF NANO STRUCTURES & COMPOSITES AT SUB-30 NM RESOLUTION, WITH A NOVEL LAB BASED X-RAY MICROSCOPE

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Abstract

In this article we describe a 3D x-ray microscope based on a laboratory x-ray source operating at 2.7, 5.4 or 8.0 keV hard x-ray energies. X-ray computed tomography (XCT) is used to obtain detailed 3D structural information inside optically opaque materials with sub-30 nm resolution. Applications include imaging internal 3D arrays of nanostructures of smart materials, polymer nanocomposites, porosity and structural imaging within fuel cells; understanding the internal workings of nanosensors, imaging of whole hydrated cells and tissues; non destructive reverse engineering and failure analysis of semiconductor circuitry and MEMs devices.

Introduction

High resolution x-ray imaging techniques have a major impact on nanoscale research by enabling fundamentally new ways of characterizing nanoscale materials. They can be used to obtain detailed 3D characterization of the composition, chemistry, texture, structure and defects near and within nanoscale features. A significant advantage over other techniques is the minimal sample preparation needed, resulting in intact specimens which can be used for in-situ characterization.

Most high resolution x-ray microscopes operate on synchrotron radiation facilities [1-6]. Even though the resulting research have advanced our understanding in many scientific fields, access to such facilities is still very limited.

We describe a novel lab based x-ray microscope[7-8] using hard x-rays operating at 2.7, 5.4 or 8 keV. Applications include non-destructive, nanoscale CT (computer tomography) for a diverse range of materials that are of interest to the military and nanotechnology community.

1.1 Optics and System configuration

Unlike most of the x-ray microscopes installed at synchrotron facilities which use soft x-ray energies[1-5], the Xradia laboratory system uses hard x-ray energies from a laboratory source. Conventional laboratory x-ray tubes, with target materials such as Rh, Cr and Cu, generate photon energies at 2.7, 5.4 and 8.0 KeV respectively, may be used in this setup.

Some of the advantages of using x-rays with higher energies are to provide greater penetrating power and depth of focus, making it possible to image thicker specimens which are advantageous for inorganic, materials, such as semiconductor, ceramics and metals. For example, the sample thickness requirement for tomography using soft x-rays is typically 1 to 2 μm for most materials, but at the higher x-ray energy, say at 8 keV, the sample thickness requirement can be relaxed to 100 μm. The imaging optics of our x-ray microscope are Fresnel zone plates[9] (Figure 1) and x-ray optical components fabricated by Xradia.

Fresnel zone plates are diffractive optical elements which focus x-rays by means of diffraction. Spatial resolution is determined by the width of the outermost zone width Δr_n, while the efficiency is a function of the plate. Depth of field is a function of Δr_n and x-ray energy. Ultimately, the main challenge for hard x-ray microscope is to develop zone plates with highest possible aspect ratio between the thickness and the outermost zone width of the zone plate.

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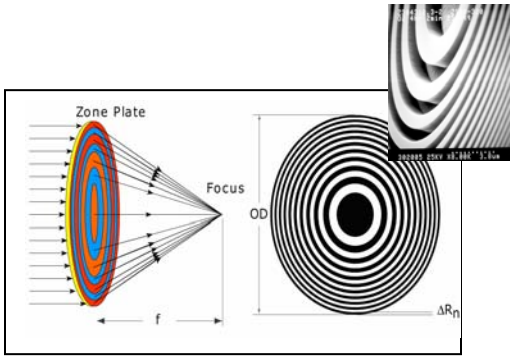

Figure 1: Fresnel Zone plates as focusing optics for x-rays. The Rayleigh resolution δ of a x-ray microscope is proportional to the outermost zone width Δr_n of the zone plate: $\delta = 1.22\Delta r_n$

The Xradia x-ray microscope is configured like a typical full field microscope incorporating a source, condenser and objective optics as shown in (Figure 2).

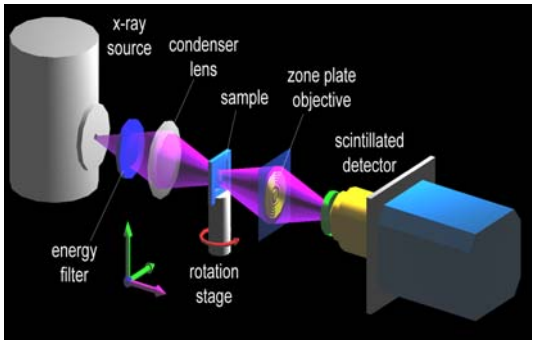

Figure 2: Schematic of nanoXCT 3D Xray microscope with specialized Fresnel Zone plate optics, condensers for high resolution applications with a nano-precision sample stage.

Contrast in transmission x-ray imaging is mainly based on absorption differences between different materials within the sample. For most materials encountered in nanotechnology such as biological, polymer, composites and semiconductor, the attenuation length differences between materials can be very small, resulting in little or no contrast in the image. To overcome this problem, the Xradia x-ray microscope can operate in the Zernike phase contrast mode by incorporating a phase ring in the back focal plane of the zone plate[9-10]. The phase ring phase-shifts the rays of the undiffracted beam, while the phase of the rays diffracted by the sample remain unchanged. Zernike phase contrast imaging can significantly improve contrast for most materials.

By rotating the specimen on an ultra precision stage, tomographic images with a 20 um × 20 um field of view, and specimen up to 100 um thick can be obtained at 40 nm resolution. This configuration is applicable to optically opaque materials ranging from semiconductor chips to ceramic fuel cells. Soft materials such as biological cells and organic materials including polymers and composites can be imaged at 2.7 keV with sufficient penetration power. Since high-resolution zone plates are easier to fabricate for lower x-ray energies due to relaxed requirement for zone plate aspect ratio, sub-30 nm resolution has been demonstrated with the Xradia x-ray microscope.

1.2 Applications to the Military and Nanotechnology

The 3D x-ray microscope is suitable for non-destructive characterizing of diverse range of materials. This includes imaging 3D nanostructure arrays of smart materials for lightweight uniform and armor, polymer nanocomposites for various combat applications or porosity and structural imaging within fuel cells. Furthermore understanding the internal workings of nanosensors, imaging of whole hydrated cells and tissues, non destructive reverse engineering and failure analysis of semiconductor circuitry and MEMs have been investigated. The examples below (Figures 3-5) include failure analysis, competitive analysis of components, and DVL (device versus layout) verification of manufacturing integrity by military subcontractors of semiconductor devices and chips.

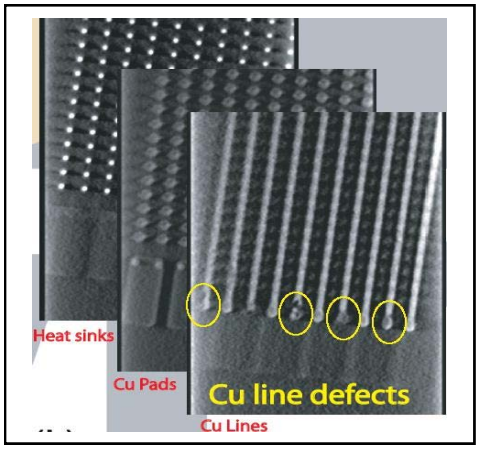

Figure 3: Failure analysis showing buried Cu line defects in ICs without mechanical/chemical deprocessing. Imaged with 5.4 keV x-ray

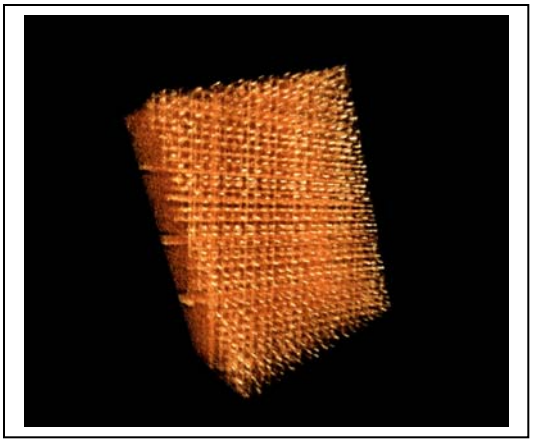

Figure 4: 3D x-ray rendered image of a Pentium IV chip, with 9 metal layer stack. 8 keV has sufficient energy to penetrate the entire metal stack of a 100 µm thick sample.

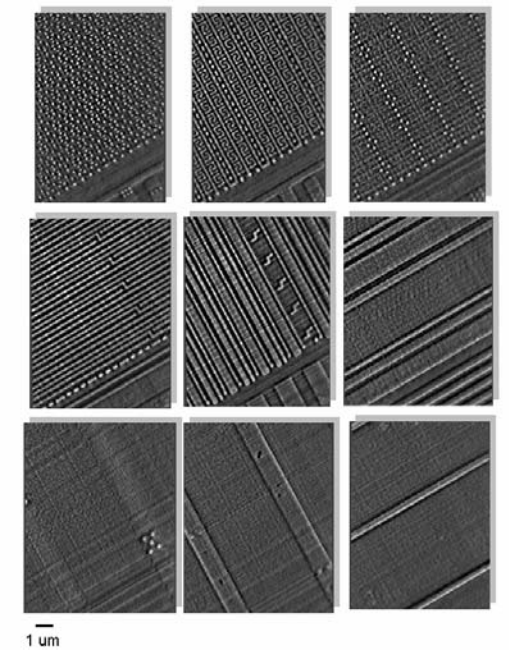

Figure 5: Just as in medical CTs, virtual cross sectioning or slicing of the sample at different planes is possible. CT slices showed the 9 metal layers in the Pentium 4 chip without the need for physical deprocessing. M1 (top row left image) to M9 (bottom row right image). Sub-100 nm voids in broad Cu lines (bottom row center image) can be observed. Sample Imaged at 8.0 keV

Imaging of biological and polymer based nanocomposites by charged particles microscopy, like TEM, is limited to very thin samples (typically << 0.5 micron). Higher Z materials such as ceramic, metals and semiconductors are typically thinned to less than 30 nm to ensure sufficient transmission of electrons making it extremely cumbersome and difficult to study the 3D array of internal structures of most novel materials.

X-rays have the advantage of deep penetration for nondestructive 3D imaging of relatively large and thick specimens. Up to 100 microns thick nano-fibers, composites, nano-sensors, fuel cell membrane (figure 6), entire multi metal layer stack within a semiconductor chip (figure 3-5), single biological cell or a cell clusters and tissues may be studied with little or no sample preparation. Samples thicker than 100 microns may be imaged in 3D, albeit at the expense of depth of focus, resolution and throughput.

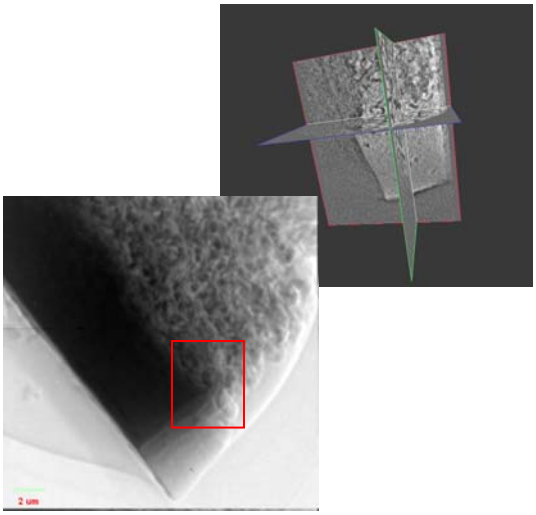

Figure 6: Solid Oxide Fuel cell (SOFC)- a potential large scale alternative energy source for the military and industry, is imaged in 3D to reveal the outer YSZ layer and internal Ni-YSZ porous structures. CT slice shows the porous-non porous phase distribution. Analysis of pore sizes, with the potential to observe structural changes in situ during operation of the cell may be implemented
Sample Courtesy : W Chiu, Univ. Connecticut, Adaptive Materials Inc

Optical and confocal microscopy suffers from diffraction limited spatial resolution due to the wavelength of visible light. Therefore, spatial resolution is generally no better than 200 nm.

Unlike Scanning Electron Microscopes (SEMs) or Transmission Electron Microscopes

(TEMs), the Xradia nanoXCT non-destructive imaging technique requires minimal preparation. It also does not require a vacuum environment nor an electrically conductive sample. Surfaces and buried structures can be imaged at sub-30 nm resolution.

Techniques, such as scanning near-field optical microscopy, scanning electron microscopy, and atomic force microscopy (AFM) are typically limited to image surface structures. While the internal 3D architecture of structures, pores or laminar arrays can be characterized by these surface techniques by making cross sections or by microtomy, these preparation processes are tedious, creates artifacts and in certain cases, are physically impossible, for example, with materials which are very hard, brittle, elastic, soft or wet.

2.2 Additional advantages of x-rays

X-rays offer several other unique intrinsic advantages for characterizing a wide range of novel materials, from high to low Z (including biological, polymer nanostructures and composites) :

- Extendibility. Short wavelength of hard x-rays makes high resolution imaging of relatively thick materials possible. Molecular length scales, with sub-30nm is currently achieved with specialized x-ray optics for hard x-rays from Xradia. The system can be extendable for higher resolution, as and when higher resolution zone plate fabrication technology advances, which currently is about 25 nm for 2.7 keV energy. Spatial resolution achievable for x-ray microscopy in the near future is possibly around 10 nm.

- Rich contrast mechanisms can be exploited with x-ray microscopy, ranging from absorption contrast, elemental contrast, phase contrast mechanism (figure 7) and function-specific labeling techniques in cellular research in biology.

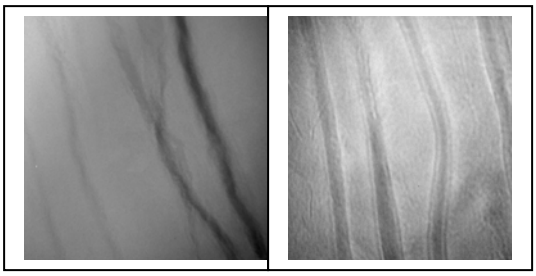

Fig 7. Dentin sample with normal absorption contrast(left) and Zenike phase contrast, showing additional fine details (right). Images acquired at 5.4 keV. Sample courtesy: Dr. J. Kinney, LLNL

- Because of the longer focal length associated with hard x-ray microscopy, there is generally sufficient working distance between the x-ray source and sample, making it possible to install fixtures for in-situ experiments. In-situ or time-resolved studies of material changes under simulated or real conditions such as stress migrations, structural deformation, dynamic interfacial interactions or structural changes under dry, humid, aqueous or temperature controlled environments, can be implemented.

Conclusion

Laboratory based 3D x-ray microscopy offers a complementary and versatile high-resolution imaging tool for non-destructive imaging and characterization of a host of novel nanomaterials currently under active investigation by the military and industry. It bridges the gap between optical microscopy and those of electron microscopy- with a compelling advantage as a minimally invasive technique, requiring little or no sample preparation. The system can be configured to image samples in its natural state or operating conditions- thereby helping researchers to shorten the developmental time line or time to market.

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